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## EUROPEAN PATENT APPLICATION

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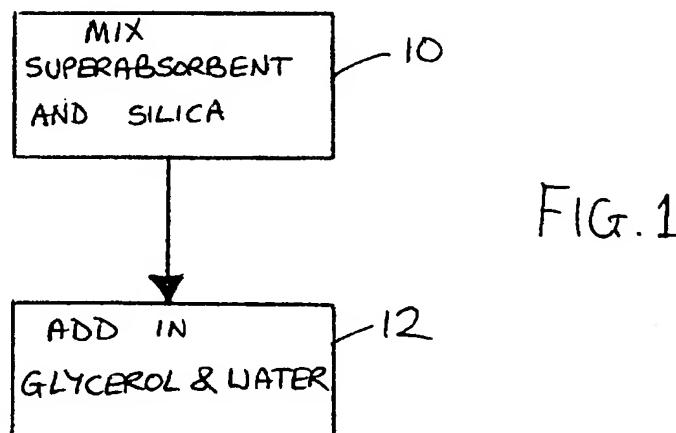
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### (54) Absorbent material and method of production

(57) A superabsorbent formulation is described for absorbing liquids (for example in an ostomy, incontinence or woundcare application), the formulation comprising superabsorbent (100 pbw), silica (3-20 pbw), water (0.5-8 pbw) and glycerol (5-40 pbw). The superabsorbent and silica are mixed together in a first production

stage (10), to coat the superabsorbent particles with silica. The glycerol and water are added during a second production stage (12). The effect of the silica is to increase the amount of glycerol and water which can be taken up in the formulation, thereby producing a product with greater structural stability when consolidated (compressed).



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**Description**

[0001] This invention relates to an absorbent material, and to a method of producing the material. The invention is especially suitable for absorbing liquid matter excreted by the body (e.g. for use in ostomy, incontinence or woundcare), and/or for absorbing aqueous matter, but it is not limited exclusively to these fields.

[0002] GB-A-2301350 and EP-A-0891758 describe superabsorbent formulations and methods of producing articles containing superabsorbent material. A significant aspect of the superabsorbent formulation is the inclusion of glycerol and water, which binds the superabsorbent powder granules together when compressed, to form a consolidated mass. The superabsorbent material can be formed into tablets, or compressed between upper and lower layers to form a sheet-like product.

[0003] However, there are practical limits on the amount of glycerol and water which the superabsorbent is able to take up as a consolidatable mass. This limits the structural stability of the consolidated mass, and makes it vulnerable to at least some degree of shedding (particularly at the edges). Also, articles made of the material tend not to be flexible, and may shed material if bent or flexed.

[0004] It would be desirable to provide a way of increasing the amount of glycerol and water which can be incorporated in the formulation without detracting significantly from other advantageous characteristics of the superabsorbent formulation.

[0005] The present invention has been devised bearing the above in mind.

[0006] Broadly speaking, one aspect of the invention is to include silica in a formulation including superabsorbent material (e.g. powder or granules). This has been found to provide the following advantages:

(a) the silica acts as a lubricant, enabling the superabsorbent particles to flow more easily, thus making handling of the superabsorbent particles easier, and leading to easier mixing and compressing processes; and

(b) the silica also enables more glycerol/water to be taken up by the superabsorbent material. It is believed that the silica enables the glycerol/water to penetrate between the particles of superabsorbent material more completely, thereby increasing the quantity of glycerol/water which can be taken up.

[0007] Preferably, the amount of silica present (per 100 parts wt of superabsorbent material) is at least 3 parts, more preferably at least 4 parts, more preferably at least 5 parts, more preferably at least 6 parts, more preferably at least 7 parts, and in one form at least 8 parts.

[0008] Preferably, the amount of silica present (per 100 parts wt of superabsorbent material) is not greater

than 20 parts, more preferably not greater than 15 parts, more preferably not greater than 10 parts and, in one form, not greater than 8 parts.

[0009] The optimum amount of silica depends to some extent on the size of the particles of superabsorbent. Generally, it is preferred that the silica be present in such a quantity to provide a surface coating on substantially all of the superabsorbent particles. Preferably, each particle is substantially entirely coated. It will be appreciated that a smaller particle size results in a larger surface area to be coated per unit volume, thus requiring a greater quantity of silica for optimum effect.

[0010] The superabsorbent particles may comprise an alkali metal polyacrylate (e.g. sodium polyacrylate), or some other polyacrylate, or other material which provides superabsorbent properties (defined generally as the ability to absorb many times their own weight of liquid matter). Suitable materials include for example, water-swellable polymers.

[0011] Preferably, the formulation includes glycerol. Alternatively, another non-volatile lubricious polyhydroxy compound may be used which is water-soluble or water-dispersible at or below 40°C and is liquid at room temperature (15 to 20°C). Suitable other compounds include, for example: propane 1,2 diol; polyethylene glycol 200 (or 300); or sorbitol.

[0012] Preferably, the amount of glycerol (or other material) is 5-40 parts per 100 parts of the superabsorbent material, more preferably 5-30 parts, more preferably 10-25 parts. In one embodiment, the amount of glycerol (or other material) is between 12 and 18 parts per 100 parts of superabsorbent.

[0013] Preferably, the formulation also includes water. Preferably, the water is present in an amount of 0.5 to 10 parts by weight per 100 parts of superabsorbent, more preferably 0.5 to 6 parts. In one example, the amount of water is between 3 and 5 parts.

[0014] The formulation may also include additives, for example, a malodour counteractant.

[0015] In another aspect, the invention provides a method of producing an absorbent material, the method comprising:

(a) mixing together superabsorbent particles and silica; and  
(b) adding to the mixture from step (a) a cohesive formulation to make the mixture consolidatable.

[0016] Preferably, the silica and superabsorbent particles are mixed to provide a mixture in which substantially all of the superabsorbent particles are coated with silica. Preferably, the particles are substantially entirely coated with silica.

[0017] Preferably, the cohesive formulation is added after the mixing in step (a) is carried out.

[0018] Preferably, the cohesive formulation includes glycerol and water. Instead of glycerol may be used another non-volatile lubricious polyhydroxy compound

which is water-soluble or water-dispersible at or below 40°C and is liquid at room temperature (15 to 20°C).

[0019] An embodiment of the invention is now described by way of example only, with reference to the accompanying drawings, in which:

Fig. 1 is a schematic flow diagram showing a production process for producing an absorbent material;

Figs. 2a and 2b are schematic drawings showing the superabsorbent granules before (a) and after (b) mixing with silica; and

Fig. 3 is a schematic drawing of apparatus for consolidating the material to form shaped articles.

[0020] An example superabsorbent formulation comprises, in parts by weight (pbw):

Superabsorbent	100 pbw
Silica	6-10 pbw
Glycerol	14 pbw
Water	4 pbw.

[0021] A suitable superabsorbent material is, for example, sodium polyacrylate available under trade name FemDry 33C from Chemdal. Sodium polyacrylate superabsorbent materials are also available from Dow Chemicals and from Hoescht.

[0022] Referring to Figs. 1 and 2, the formulation is prepared in first and second stages 10 and 12. The first stage is to mix together the superabsorbent granules and the silica. Fig. 2a shows the superabsorbent granules 14 prior to mixing, and Fig. 2b shows the granules 14 after mixing. As can be seen from Fig. 2b, the effect of mixing is to coat the granules 14 with a thin coating of silica 16. The silica 16 acts as a lubricant making the granules easier to flow, and therefore easier to mix.

[0023] The second stage 12 is to add a premix of glycerol and water to the silica coated superabsorbent formulation. The effect of the silica is generally to enable the superabsorbent formulation to take up a greater quantity of glycerol/water than if the silica were absent. It is believed that this is as a result of the silica (again acting as a form of lubricant) permitting the glycerol/water to penetrate more completely between the superabsorbent granules 14. The optimum quantity of silica depends at least to some extent on the size of the superabsorbent granules 14. It is preferred that the quantity of silica be sufficient to coat substantially the entire surface of the superabsorbent particles 14. During the second stage 12, the formulation is mixed to a powder consistency.

[0024] The superabsorbent formulation can be formed into a desired shaped article by compression, which consolidates the powder structurally, in the same manner as that described in GB-A-2301350 and EP-A-0891758 referred to hereinbefore. The presence of the

glycerol/water (particularly in the higher quantity than that attainable hitherto) enables the mixture to be compressed at room temperature with only relatively mild pressure being required. In most cases no other heat is

5 required. Moreover, the increased quantity of glycerol/water provides greater structural stability than that achievable hitherto, so that the formed article is less prone to shedding, and can be at least partly flexible if desired.

[0025] Fig. 3 illustrates, by way of example, apparatus 18 for forming a sheet of superabsorbent, suitable for use in ostomy, incontinence or woundcare applications. The powder formulation is fed from a supply 20 onto a travelling sheet 21 of tissue paper which is pulled off a

15 supply roll 22. A second (overhead) sheet 23 of tissue paper is fed from a second supply roll 24 and these two sheets sandwich the powder between them. The product passes between a first pair of rolls 26 which form a first nip 27 and to a second pair of rolls 28 forming a

20 second nip 29. The inter-roll spacing at the second nip 29 may be for example 0.1 to 2.0 mm. That at the first nip 27 may be 0.75 to 4 mm. No external heat is applied. The resulting product is a flat sheet which can then be cut into suitable shapes, e.g. rectangles 32, and can be 25 directly placed in an ostomy pouch or incontinence pouch. It has been found to rapidly absorb a liquid such as urine. In tests, over 60 ml of synthetic urine were absorbed in under 60 seconds, usually under 40 seconds, by a product of one square centimetre area and 2 mm thickness made by the method described above.

[0026] If desired, the sheet may be processed during production to form packet-like articles, as described in EP-A-0891758.

[0027] Reference has been made in the above specification to malodour counteractants. One example of a malodour counteractant is a bactericide, e.g. benzyl alkonium chloride. Another example is a fragrance. Other examples are included in a range of bactericides manufactured by ConvaTec™ (Calgon-Vestal Div.). Yet 30 other malodour counteractants which may be used are methylchlorosothiazolinone, methylisothiazolinone, or denatonium benzoate.

[0028] It will be appreciated that the foregoing description is merely illustrative of preferred forms of the 45 invention, and that many modification may be made within the scope of the invention. Aspects believed to be of particular importance are identified in the claims. However, the applicant claims protection for any novel feature or idea described and/or illustrated herein 50 whether or not emphasis has been placed thereon.

## Claims

55 1. A formulation for utilisation to absorb liquid, comprising:  
superabsorbent;

silica;  
water; and  
a non-volatile lubricious polyhydroxy compound which is water-soluble or water-dispersible at or below 40°C and is liquid at room temperature (i.e. at at least one temperature within the range 15 to 20°C) 5 to 40 pbw.

2. A formulation according to claim 1, wherein relative quantities in the formulation are, in parts by weight (pbw):

superabsorbent	100 pbw
silica	3-20 pbw
water	0.5-8 pbw
said lubricious polyhydroxy compound	5-40 pbw.

3. A formulation according to claim 2, wherein the silica is present in a quantity to provide a surface coating on at least the majority of the surface area of the superabsorbent.

4. A formulation according to claim 3, wherein the silica is present in a quantity to provide a surface coating on substantially the entire surface area of the superabsorbent.

5. A formulation according to 2 or any claim dependent thereon wherein the silica is present in a quantity of at least 4 pbw.

6. A formulation according to claim 5, wherein the silica is present in a quantity of at least 5 pbw.

7. A formulation according to claim 6, wherein the silica is present in a quantity of at least 6 pbw.

8. A formulation according to claim 7, wherein the silica is present in a quantity of at least 7 pbw.

9. A formulation according to claim 8, wherein the silica is present in a quantity of at least 8 pbw.

10. A formulation according to claim 2, or any claim dependent thereon, wherein the silica is present in a quantity of not greater than 20 pbw.

11. A formulation according to any preceding claim wherein the polyhydroxy compound is glycerol.

12. A formulation according to claim 2, or any claim dependent thereon, wherein the polyhydroxy compound is present in an amount of 5-30 pbw.

13. A formulation according to claim 2, or any claim dependent thereon, wherein the polyhydroxy compound is present in an amount of at least 12 pbw.

14. A formulation according to claim 13, wherein the polyhydroxy compound is present in an amount of at least 13 pbw.

15. A formulation according to claim 14, wherein the polyhydroxy compound is present in an amount of at least 14 pbw.

16. A formulation according to claim 2, or any claim dependent thereon, wherein the water is present in an amount of 0.5-6 pbw.

17. A formulation according to claim 2, or any claim dependent thereon, wherein the water is present in an amount of at least 3 pbw.

18. A formulation according to claim 17, wherein the water is present in an amount of at least 4 pbw.

19. A formulation according to any preceding claim, wherein the superabsorbent comprises a water swellable polymer.

20. A formulation according to claim 19, wherein the superabsorbent comprises a polyacrylate.

21. A formulation according to claim 20, wherein the polyacrylate is an alkali metal polyacrylate.

22. A formulation according to claim 21, wherein the superabsorbent comprises sodium polyacrylate.

23. A method of producing a superabsorbent formulation, the method comprising:

(a) mixing together superabsorbent and silica to coat the superabsorbent with silica; and  
(b) mixing with the silica coated superabsorbent, water and a non-volatile lubricious polyhydroxy compound which is water-soluble or water-dispersible at or below 40 °C and is liquid at room temperature (i.e. at at least one temperature within the range 15 to 20°C).

24. A method according to claim 23, wherein the lubricious polyhydroxy compound is glycerol.

25. A method according to claim 23 or 24, wherein the relative quantities in the formulation are, in parts by weight (pbw):

superabsorbent	100 pbw
silica	3-20 pbw
water	0.5-8 pbw
said lubricious polyhydroxy compound	5-40 pbw.

26. A method according to claim 23, 24 or 25, wherein

the silica is present in a quantity to provide a surface coating over substantially the majority of the surface area of the superabsorbent.

27. A method according to claim 26, wherein the silica is present in a quantity to provide a surface coating on substantially the entire surface area of the superabsorbent. 5
28. A method of producing a superabsorbent formulation, the method comprising: 10
  - (a) providing superabsorbent particles which are substantially entirely coated with silica; and
  - (b) mixing with the silica coated superabsorbent, water and a non-volatile lubricious polyhydroxy compound which is water-soluble or water-dispersible at or below 40°C and is liquid at room temperature (i.e. at at least one temperature in the range of 15°C-20°C). 15 20
29. An ostomy, incontinence or woundcare product comprising or containing a superabsorbent formulation as defined in any of claims 1 to 22. 25
30. An ostomy product according to claim 29, wherein the product is an ostomy pouch.
31. Use of a superabsorbent formulation as defined in any of claims 1 to 22 in an ostomy, incontinence or woundcare product. 30

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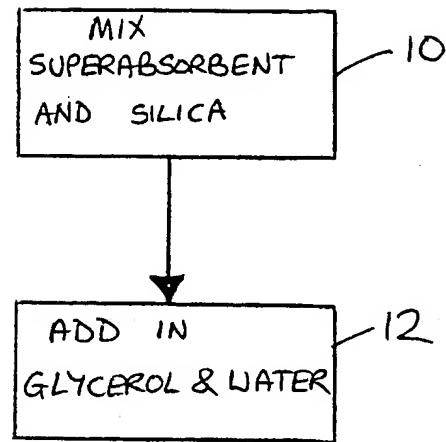


FIG. 1

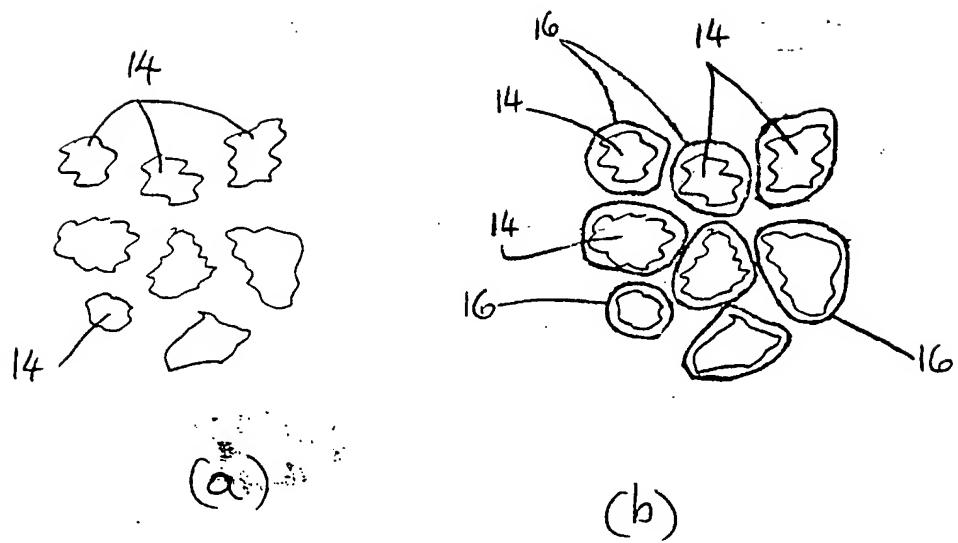


FIG. 2

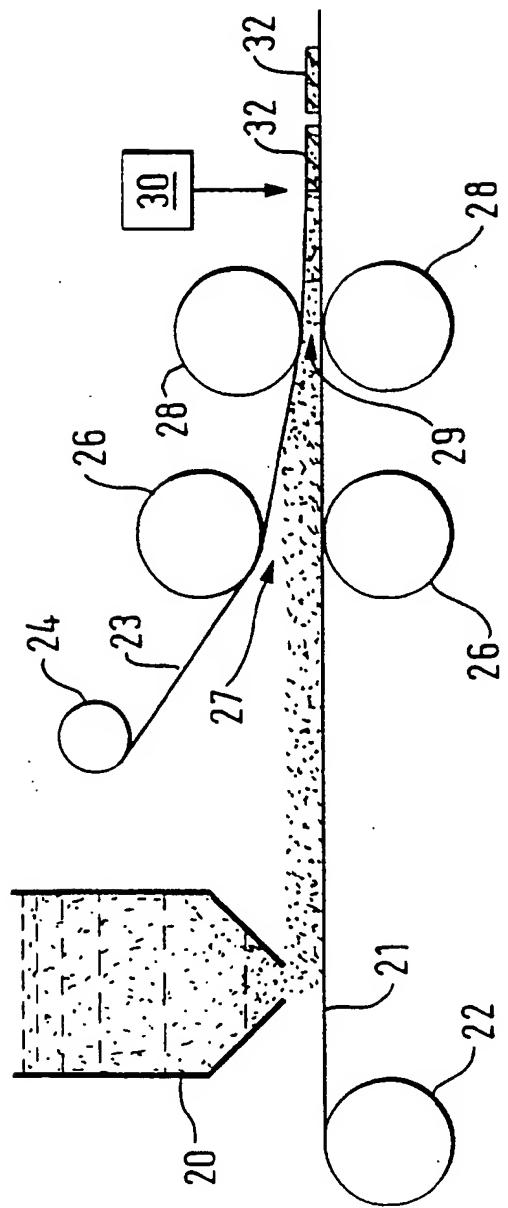


FIG. 3



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## EUROPEAN SEARCH REPORT

Application Number  
EP 00 30 3141

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.)
A	EP 0 138 427 A (PERSONAL PRODUCTS CO) 24 April 1985 (1985-04-24) * page 6, line 32 - page 7, line 28 * ----	1-31	A61L15/60 A61L15/18 A61L28/00
A	GB 2 312 213 A (MINNESOTA MINING & MFG) 22 October 1997 (1997-10-22) * page 2, line 7 - line 28 * * page 4, line 6 - line 25 * * examples * ----	1-31	
A	US 5 489 469 A (KOBAYASHI TAKATOSHI ET AL) 6 February 1996 (1996-02-06) * column 2, line 14 - line 22 * * column 6, line 31 - line 43 * ----	1-31	
A	EP 0 629 411 A (SANYO CHEMICAL IND LTD) 21 December 1994 (1994-12-21) * page 15, line 46 - page 16, line 21 * ----	1-31	
A,D	GB 2 301 350 A (SQUIBB BRISTOL MYERS CO) 4 December 1996 (1996-12-04) ----		TECHNICAL FIELDS SEARCHED (Int.Cl.)
A,D	EP 0 891 758 A (SQUIBB BRISTOL MYERS CO) 20 January 1999 (1999-01-20) ----		A61L
<p>The present search report has been drawn up for all claims</p>			
Place of search	Date of completion of the search	Examiner	
THE HAGUE	14 September 2000	Cousins-Van Steen, G	
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14-09-2000

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
EP 0138427	A	24-04-1985	AT 50696 T AU 578042 B AU 3341084 A BR 8404688 A CA 1236074 A DE 3481480 D DK 453984 A ES 536164 D ES 8600962 A FI 843719 A GR 80377 A HK 50090 A JP 60099339 A KR 9105291 B NO 843800 A NZ 209483 A PT 79248 A,B SG 61190 G ZA 8407471 A	15-03-1990 13-10-1988 28-03-1985 13-08-1985 03-05-1988 12-04-1990 24-03-1985 01-11-1985 16-02-1986 24-03-1985 15-11-1984 08-07-1990 03-06-1985 24-07-1991 25-03-1985 31-08-1987 01-10-1984 07-09-1990 28-05-1986
GB 2312213	A	22-10-1997	EP 0897304 A WO 9738740 A	24-02-1999 23-10-1997
US 5489469	A	06-02-1996	JP 1990709 C JP 7020548 B JP 63185447 A JP 7028891 B JP 63246159 A JP 1990716 C JP 7020549 B JP 63267435 A CA 1314698 A CN 88100333 A,B DE 3875749 A DE 3875749 T EP 0278601 A ES 2035259 T HK 50194 A KR 9108362 B PH 25432 A SG 59394 G	08-11-1995 08-03-1995 01-08-1988 05-04-1995 13-10-1988 08-11-1995 08-03-1995 04-11-1988 23-03-1993 24-08-1988 17-12-1992 10-02-2000 17-08-1988 16-04-1993 27-05-1994 12-10-1991 01-07-1991 28-10-1994
EP 0629411	A	21-12-1994	FI 942915 A JP 7088171 A	19-12-1994 04-04-1995
GB 2301350	A	04-12-1996	NONE	

EPO FORM R459  
For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.

EP 00 30 3141

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

14-09-2000

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP 0891758	A 20-01-1999	GB	2325432 A	25-11-1998
		AU	6601398 A	26-11-1998
		CA	2238248 A	21-11-1998
		JP	11020058 A	26-01-1999
		US	6054631 A	25-04-2000